13946

8TH INTERNATIONAL CONFERENCE ON STABILITY AND HANDLING OF LIQUID FUELS
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BIODIESEL AND BIODIESEL BLEND PROPERTIES RELATED TO EPACT USE

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Biodiesel is defined as "a fuel composed of mono-alkyl esters of long chain fatty acids derived from vegetable oil or animal fats, designated B100" in the American Society for Testing and Materials (ASTM) D 6751 specification for Biodiesel Fuel (B100) Blend Stock for Distillate Fuels. In 1992, the U.S. Congress enacted the Energy Policy Act (EPAct) requiring federal and state vehicle fleets to purchase alternative fueled vehicles (AFV). EPAct was amended in 1998 as the Energy Conservation and Reauthorization Act (ECRA) to include biodiesel as an option for meeting AFV acquisition requirements by purchasing and using either 450 gallons of biodiesel or 2250 gallons of biodiesel blend consisting of 20% volume biodiesel in petroleum diesel fuel (designated B20). An effort initiated by U.S. Army TACOM/TARDEC/NAC to provide a specification for B20 biodiesel blended fuel for use by Government agencies resulted in publication of Commercial Item Description (CID) A-A-59693, Diesel Fuel, Biodiesel Blend (B20). This report summarizes data developed in a project designed to characterize selected biodiesel samples (identified in market survey, TARDEC Technical Report No. 13801) and biodiesel (B20) blends made with diesel fuels. The biodiesel feed stocks included unused soybean oil, used cooking oil, used soybean cooking oil, unused vegetable oil, used vegetable oil, unused canola oil, unused cottonseed oil, and yellow grease. Various chemical and physical properties were determined to ensure compliance with B20 and B100 specification requirements. The data was instrumental in deciding that the B20 specification should be restricted to one grade of biodiesel blend as the winter grade low sulfur diesel fuel No. 1-D (LS 1-D) has too restrictive of a distillation requirement. The B100 samples were also tested for oxidation stability in accordance with ASTM D 6186 at 125°C, using pressure differential scanning calorimetry (PDSC). The biodiesels having the highest level of unsaturation were the most reactive but ranged considerably depending on source. Biodiesel from used feedstock and all B20 blends did not have measurable induction times at the selected test temperature and are considered to be more oxidatively stable than the soy based biodiesels that had measurable induction times.

BACKGROUND

Alternative fuels made from materials other than petroleum are available for use in alternative fueled vehicles (AFVs) and some conventional vehicles. Liquid fuels such as biodiesel blends could be used in non-tactical U.S. Army or other Military/Federal Government compression ignition (CI) engine powered vehicles. The military combat/tactical fleet is exempt from Federal Government mandates to use alternative fueled vehicles and has adopted JP-8/JP-5 jet fuel as the primary military fuel. The Army non-tactical fleet and other Federal nonexempt CI engine powered vehicles are possible candidates for using biodiesel. Inclusion of biodiesel as an alternative fuel qualifying for alternative fueled vehicle credits for fleets required to meet AFV requirements have allowed for its use at 20 (minimum) percent biodiesel in petroleum diesel fuel (B20). Alternative fuels are being considered for the 21st Century Truck (21T) program. [1]*

*Numbers in brackets refer to references.

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14. ABSTRACT

Biodiesel is defined as "a fuel composed of mono-alkyl esters of long chain fatty acids derived from vegetable oil or animal fats, designated B1 00" in the American Society for Testing and Materials (ASTM) D 6751 specification for Biodiesel Fuel (B100) Blend Stock for Distillate Fuels. In 1992, the U.S. Congress enacted the Energy Policy Act (EPAct) requiring federal and state vehicle fleets to purchase alternative fueled vehicles (AFV). EPAct was amended in 1998 as the Energy Conservation and Reauthorization Act (ECRA) to include biodiesel as an option for meeting AFV acquisition requirements by purchasing and using either 450 gallons of biodiesel or 2250 gallons of biodiesel blend consisting of 20% volume biodiesel in petroleum diesel fuel (designated B20). An effort initiated by U.S. Army TACOM/TARDEC/NAC to provide a specification for B20 biodiesel blended fuel for use by Government agencies resulted in publication of Commercial Item Description (CID) A-A-59693, Diesel Fuel, Biodiesel Blend (B20). This report summarizes data developed in a project designed to characterize selected biodiesel samples (identified in market survey, TARDEC Technical Report No. 13801) and biodiesel (B20) blends made with diesel fuels. The biodiesel feed stocks included unused soybean oil, used cooking oil, used soybean cooking oil, unused vegetable oil, used vegetable oil, unused canola oil, unused cottonseed oil, and yellow grease. Various chemical and physical properties were determined to ensure compliance with B20 and B100 specification requirements. The data was instrumental in deciding that the B20 specification should be restricted to one grade of biodiesel blend as the winter grade low sulfur diesel fuel No. 1-D (LS 1-D) has too restrictive of a distillation requirement. The B100 samples were also tested for oxidation stability in accordance with ASTM D 6186 at 125?C, using pressure differential scanning calorimetry (PDSC). The biodiesels having the highest level of unsaturation were the most reactive but ranged considerably depending on source. Biodiesel from used feedstock and all B20 blends did not have measurable induction times at the selected test temperature and are considered to be more oxidatively stable than the soy based biodiesels that had measurable induction times.

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In order to facilitate the purchase of B20, U.S. Army TACOM-TARDEC, in its role as the DOD Executive Agent for ground fuels and lubricants as per Army Regulation 70-12, has issued a Commercial Item Description (CID) for B20 with designation A-A-59693. This was done in coordination with several Government agencies (such as DOD, DESC, NREL, and USDA) and Industry (such as Engine OEMs, the National Biodiesel Board, biodiesel manufacturers and suppliers, research institutes, etc.). [2]

The General Services Administration has authorized the use of CID A-A-59693 (for B20 biodiesel fuel blends), which relies upon each fuel component meeting their own specific specification requirement. In other words, diesel fuel used in blending B20 must meet the ASTM D 975 industry standard requirements and the neat biodiesel must meet ASTM D 6751 requirements. In addition, other requirements were placed upon the finished B20 in order to ensure satisfactory product quality and engine operability. These additional requirements were:

(a) Fuel Appearance (ASTM D 4176): The B20 shall be visually free from undissolved water, sediment and suspended matter.
(b) Acid Number (ASTM D 664): 0.25 mg KOH/g sample maximum requirement.
(c) Particulate Contamination (ASTM D 6217): 10 mg/L maximum requirement.
(d) Free Water (ASTM D 2709): 0.05 volume percent maximum requirement.
(e) Low Temperature Properties: Manufacturers can meet either the Cloud Point or the Cold Filter Plugging Point. The maximum cloud point (ASTM D 2500) must be at or below the 10th Percentile Minimum Temperature for the region and season where the fuel is to be used. The maximum cold filter plugging point (ASTM D 6371) shall be at least 10°C below the cloud point requirement.

Additional B20 requirements recommended include volume percent biodiesel in B20, flash point (ASTM D 93), kinematic viscosity (at 40°C; ASTM D 445), distillation (T90; ASTM D 86), micro carbon residue (ASTM D 4530), and cetane number (ASTM D 613).

It is also noted in A-A-59693:

- Biodiesel (i.e., 100 percent biodiesel or B100) is a good solvent. Use of B20 biodiesel fuel blend may clean the fueling system of existing deposits. Users should be prepared to change fuel filters more frequently upon initial use.

- The user must be aware that a B20 biodiesel fuel blend using low sulfur diesel fuel grade 1-D as base fuel may not meet the maximum viscosity nor the maximum 90 percent recovered distillation temperature requirements for LS 1-D in A-A-52557 and ASTM D 975. The significance of this deviation has not been established.

- Original Equipment Manufacturers (OEM) biodiesel allowances for use and impact of biodiesel is available and should be checked.

INTRODUCTION

The Federal Government has taken the lead in establishing a market for AFVs by mandating their purchase across federal and state vehicle fleets. Table 1 provides a summary of Federal Energy Acts and Presidential Executive Orders related to energy use. [3-4] In addition, the government has offered incentives to automobile manufacturers to encourage the production of AFVs. The government has also established consumer awareness programs to educate the public about the benefits of AFVs. The future of AFVs is uncertain because consumer demand for them has not been established. [3] AFVs are environmentally friendly and have the potential to provide short-, mid-, and long-term solutions to the U. S. problem of dependence on foreign oil. At this time, there is little economic incentive to encourage consumers to purchase AFVs. [3]

This report addresses biodiesel fuel as an alternative fuel and ways to utilize it as a blend stock with diesel fuel. Title XII of Section 101(a) of the Omnibus Consolidated and Emergency Supplemental Appropriations Act of FY 1999 (Public Law 105-277) permits Federal Agencies to meet up to 50 percent of their AFV acquisition requirements by using biodiesel fuel.

Table 1. Federal Actions Driving Towards Alternative Fuels [1,4]

Federal Action	Date	Driving Impact Potential
Clean Air Act (CAA)	1990	The CAA, among other things, strives to reduce engine fuel exhaust emissions. As alternative fuels have less emissions than conventional petroleum fuels, CAA indirectly encourages the use of alternative fuels as these will reduce certain emissions such as SOx.
Executive Order 12759 [Federal Energy Management] (superseded by Executive Orders 12902 and 13123)	5/17/1991	Directs all Federal Agencies to develop energy efficiency goals for facilities and for vehicles, to procure energy efficient goods and products, and to procure AFVs.
Energy Policy Act of 1992 (EPACT) [Public Law 102-486]	10/24/1992	Requires the Federal Government to procure large numbers of AFVs in the coming years as a means of fostering energy security and a cleaner environment.
Executive Order 12844 [Federal Fleet Conversion Task Force] (superseded by Executive Order 13031)	4/21/1993	Tasked the Federal Agencies to exercise leadership in the use of AFVs. Each agency was required to adopt aggressive plans to substantially exceed the AFV purchase requirements that had been established by EPACT.
Executive Order 13031 [Federal Alternative Fueled Vehicle Leadership]	12/13/1996	Required each agency to develop and implement aggressive plans to fulfill the AFV acquisition requirements of the EPACT. The purpose of this order is to ensure that the Federal Government exercise leadership in the use of alternative fueled vehicles (AFVs).
Executive Order 13101 [Greening the Government Through Waste Prevention, Recycling, and Federal Acquisition]	9/14/1998	Encourages waste prevention and recycling of wastes.
Energy Conservation Reauthorization Act (ECRA) [Public Law 105-388]	11/13/1998	Modification of EPACT which allows the use of biodiesel fuel to meet requirements of federal and state fleets to purchase alternative fuel vehicles.
Executive Order 13123 [Greening the Government Through Efficient Energy Management]	6/3/1999	Encourages government agencies to expand the use of renewable energy. Agencies are also mandated to reduce the use of petroleum within their facilities. One of the ways encouraged is by switching to a less greenhouse gas-intensive, non-petroleum energy source.
Biodiesel Fuel Use Credit Interim Rule	6/18/1999	This Rule allows covered fleet to allocate AFV acquisition credits towards meeting EPACT requirements through the purchasing of biodiesel fuel.
Executive Order 13134 [Developing and Promoting Biobased Products and Bioenergy]	8/12/1999	Helps to create a powerful new research management team to focus Federal efforts with a goal of tripling U.S. use of bioenergy and bioproducts by 2010.
Executive Order 13149 [Greening the Government Through Federal Fleet and Transportation Efficiency]	4/21/2000	The purpose of this Executive Order is to ensure that the Federal Government exercises leadership in the displacement of petroleum consumption through improvements in fleet fuel efficiency and the use of alternative fuel vehicles and alternative fuels.

For the first time, fleet managers can comply with EPACT requirements by using an alternative fuel rather than acquiring additional alternative fuel vehicles. Under the new provisions, each 450 gallons of pure biodiesel (B100) used in a vehicle weighing over 8500 pounds counts as one full AFV credit. Since biodiesel, a cleaner burning alternative fuel produced from domestic renewable feedstock such as vegetable oils, is typically used as B20 (a blend of 20 percent biodiesel and 80 percent petroleum diesel) using 2250 gallons of B20 equates to one AFV credit under EPACT. [1, 3]

ARMY FUEL TYPES AND PROPERTIES

There are a number of fuels used in Army materiel. The fuels have been classified/termed as primary fuel, alternate fuel or emergency. [1] The Army has two very important documents that direct, control, and classify the fuels for use in military equipment. These are AR 70-12 (Fuels and Standardization Policy for Equipment Design, Operation, and Logistics Support) and DOD Directive Number 4140.25 (DOD Management Policy for Energy Commodities and Related Services). AR 70-12 implements DOD 4140.25. DOD 4140.25 indicates that "Primary fuel support for land-based air and ground forces in all theaters (overseas and CONUS) shall be accomplished using a single kerosene-based fuel, in order of precedence: JP-8, commercial jet fuel (with additive package), or commercial jet fuel (without additives), as approved by Combatant Commanders. Fuel support for ground forces may also be accomplished using commercially available diesel fuel when supplying jet fuel is not practicable or cost effective". It is further stated in AR 70-12 that "...all ground vehicle and equipment with compression-ignition and turbine engines will be designed to perform acceptably using kerosene-type turbine fuels such as JP-8 or JP-5, distillate fuels such as diesel fuel (CID A-A-52557)...." [2]

Following these documents, the U.S. Army has been involved in a JP-8 conversion program for the tactical/combat fleet located in Continental United States (CONUS) and Outside CONUS (OCONUS) bases. The CONUS conversion program was completed in 1999 with an expected completion for OCONUS for 2010. Already, there are a number of bases overseas converting to JP-8.

Therefore, any alternative fuels for the combat/tactical fleet must have properties that fit within the JP-8 requirements and be compatible with legacy and future systems. Non-tactical military and other federal fleets, such as trucks, buses, cars, etc. are considered potential users of alternative fuels. (See later section titled "alternative fuels and biodiesel exhaust emissions" for the DOE definition of alternative fuel.) Please note that alternate fuel is not the same as alternative fuel.

Petroleum diesel and biodiesel fuel specification properties are tabulated in Table 2 for comparison purposes.

Tenth percentile minimum ambient air temperatures for the U.S. are provided in Appendix X4, Tenth Percentile Minimum Ambient Air Temperatures, for the United States (Except Hawaii), of ASTM D 975, Standard Specification for Diesel Fuel Oils. [5] The tenth percentile minimum air temperature is defined as the lowest ambient air temperature which will not go lower on average more that 10 percent of the time. For example, the temperature ranges from a low of –49°C for Northern Alaska to a high of 3°C for Florida (south of 29° latitude) for the month of January. South East Alaska 10th percentile temperature is –19°C for the month of January. It is recognized that fuel distributors, producers, and end users in the U.S. use cloud point, wax appearance point, cold filter plugging point (CFPP), and low temperature flow test (LTFT) to estimate vehicle low temperature operability limits for diesel fuel. No independent data has been published in recent years to determine test applicability for today's fuels and vehicles. [5]

The U.S. commercial diesel specification, ASTM D 975, contains a requirement with the wording "The grades of diesel fuel oils herein specified shall be hydrocarbon oils conforming to the detailed ....." The wording in the CID A-A-52557 is more restrictive: "The fuel shall be composed of petroleum hydrocarbon fractions meeting the requirements of ASTM D 975 except as noted...."

BIODIESEL FUEL PROPERTIES

Pure biodiesel is commonly referred to as B100, while biodiesel blends are referred to as BXX where "XX" is the volume percent of biodiesel in petroleum-based diesel fuel (petrodiesel), e.g., B20 is a blend of 20 percent biodiesel and 80 percent petroleum diesel. In December 1998, the ASTM Subcommittee D.02.E (on Burner, Diesel, Non-Aviation Gas Turbine and Marine Fuels) approved a provisional standard specification for B100 as a blend stock with petroleum diesel in concentrations up to 20 percent biodiesel

(B20). The current version of ASTM D 6751 was preceded by ASTM PS 121 approved by ASTM on May 27, 1999, and published in September 1999. [5-7]

Table 2. Comparison of Diesel and Biodiesel Specification Properties

	Petroleum Diesel Fuel				Biodiesel Fuel	
Specifications	DOD Commercial Item Description		U.S. Commercial	Test Methods	U.S. Comercial	Test Methods
	A-A-52557 (1-D)	A-A-52557 (2-D)	ASTM D 975-98b		ASTM D 6751	
Property						
Total acid number, mg KOH/g					0.80 max	D 664
S, % mass	0.05 max	0.05 max	0.05 max	D 2622	0.05 max	D 2622
Ignition Quality: Cetane Property	40 min (1)	40 min (1)	40 min (1)	D 613	40 min	D 613
One of the following must be met: 1. Aromaticity, % vol 2. Cetane Index	 35 max 40 min	 35 max 40 min	 35 max 40 min	 D 1319 D 976		
Free Glycerin, % mass					0.020 max	GC
Total Glycerin, % mass					0.240 max	GC
Distillation, °C, 90% off (recovered) End point Residue, % vol	 288, max (2) NR NR	 282-338 (2) NR NR	Grade 1-D: 288, max Grade 2-D: 282-338 (2) NR NR	D 86		
Flash pt, °C , min	38	52	Grade 1-D: 38 Grade 2-D: 52	D 93	100	D 93
Cloud pt, °C , max	10^{th} percentile min. or below for region	10^{th} percentile min. or below for region	(3)	D 2500	report to customer	D 2500
Kinematic viscosity, cSt, @ 40°C (104°F)	1.3-2.4	1.9-4.1	Grade 1-D: 1.3-2.4 Grade 2-D: 1.9-4.1	D 445	1.9-6.0	D 445
Copper strip, 3 h @ 50°C (122°F), rating	No. 3 max	No. 3 max	No. 3 max	D 130	No. 3 max	D 130
Ramsbottom C residue on 10% bottoms, % mass	0.15, max	0.35, max	Grade 1-D: 0.15 max Grade 2-D: 0.35 max	D 524	0.050 max (4)	D 524
Water and sediment, % vol	0.05 max	0.05 max	0.05 max	D 2709	0.050 max	D 1796
Particulates, mg/L	10 max	10 max		D 5452		
Ash, % mass	0.01 max	0.01 max	0.01 max	D 482		
Sulfated Ash, % mass					0.020 max	D 874
Accelerated Stability, total insolubles, mg/100 mL				D 2274, D 5304		
Scuffing Load Wear, g	(3300 min (5))	(3300 min (5)		(D 6078)		

(1) Where cetane number by Test Method D 613 is not available, Test Method D4737 can be used as an approximation.
(2) When a cloud point less than -12°C is specified, the minimum flash point shall be 38°C, the minimum viscosity at 40°C shall be 1.7 mm^2/s, and the minimum 90% recovered temperature shall be waived.
(3) Tenth percentile minimum air temperatures are provided in Appendix X4 as a means of estimating expected regional temperatures. This guidance is general. Some equipment designs or operation may allow higher or require lower cloud point fuels. Appropriate low temperature operability properties should be agreed upon between the fuel supplier and purchaser for the intended use and expected ambient temperatures.
(4) The carbon residue shall be run on the 100% sample.
(5) The 3300 g minimum is applicable to a single test result. For an average of two test results, the minimum limit of 3000 g, and for a three result average the min limit is 2800 g. Lubricity requirement in VV-F-800 (Cancelled on 2 January 1996). These values are provided here for technical reference. See also reference [13].

The definition of biodiesel contained in the specification is:

> biodiesel, n. — a fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100. [5]

Similar to petroleum diesel, the specification is based on the specific chemical and physical properties needed to ensure satisfactory diesel engine operation and is not dependent on the starting material (i.e., crude oil for petrodiesel, oil or fat for biodiesel) or the specific processing techniques used to manufacture the fuel. In the case of biodiesel, the composition of the starting oils or fats are similar. They consist mainly of triglycerides where the long chain fatty acid portion of the tryglyceride, which becomes the mono-alkyl ester (biodiesel), are straight chain hydrocarbons with 16 to 18 carbons. This phenomenon makes most of the properties of biodiesel very similar, regardless of the starting oil or fat. There are differences, however, in the amount of unsaturation in the fatty acid chains, which impacts cold flow properties (unsaturated material has better cold flow properties), cetane number (saturated material has better cetane numbers), and potentially fuel stability. [6-7]

Although the production of biodiesel involves a relatively simple chemical process, there is potential for various contaminants to be present in the fuel. These contaminants include water, free glycerin, bound glycerin, alcohol, free fatty acids, soaps, catalyst, unsaponifiable matter and the products of oxidation. Small amounts of these various contaminants have been added to biodiesel and their impact on the properties and performance of the biodiesel was measured. [8] The results generally support the levels given in ASTM D 6751. The study identifies water contamination, bound glycerin, and oxidation as three areas of concern. Pure biodiesel can hold in solution up to 2.5 times more water than the specification of 0.05 vol percent. High levels of bound glycerin can cause crystallization and increased viscosity. Oxidation can produce chemical compounds that improve cetane number, but also increase the acidity of the fuel. [8]

Frame, et al., addressed the effects of biodiesel (methyl soyate) and blends of biodiesel with petrofuels on fuel system component and material compatibility, fuel storage stability, and fuel lubricity. [9] The biodiesel was found to have excellent lubricity properties and was effective at 1 volume percent (vol%) blend in improving the lubricity of Jet A-1 fuel. The following potential problem areas associated with methyl soyate use were identified: low temperature properties, storage stability, compatibility with some metals, and compatibility with nitrile rubbers. The physical properties of the elastomers were measured according to ASTM D 471, "Standard Test Method for Rubber Property-Effect of Liquids," and ASTM D 412, "Standard Test Methods for Rubber Properties in Tension." These evaluations were performed at 51.7°C for 0, 22, 70, and 694 hours. Tensile strength, hardness, swell, and elongation were determined for all specimens. Six metal specimens were stored for 6 months at 51.7°C. Visual inspection of the specimens was performed, and total acid number (TAN) was determined using ASTM D 664, "Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration." The results showed reduced swelling characteristics in many materials when introduced to biodiesel and biodiesel fuel blends.[10] Biodiesel and biodiesel fuel blends exhibited severe corrosion with copper-containing metals and high TANs with all metals evaluated. Regarding filters, particle removal efficiency remained relatively constant for the filters evaluated in neat biodiesel and biodiesel blends. Biodiesel blends may degrade some filter media, resulting in media migration. Most water removal efficiencies were acceptable; however, the coalescing process was altered. [9]

The data in Table 3 were selected from Frame, et al., for analysis in this report. [9] The biodiesel does not distill completely at atmospheric pressure and actually begins to pyrolyze as the end point is approached using ASTM D 86 distillation. Gas chromatography methods for distillation such as ASTM D 2887 are also difficult to apply to biodiesel since it is composed of 5 to 10 components, the esterified lipids from the fats and oils from which it is manufactured. Note that the 90 percent off distillation temperatures for B20 and B30 are at or near the specification limit for diesel fuel oil grade 2-D and exceed limits for diesel fuel oil grade 1-D.

Since the viscosity of biodiesel approaches the limit for petrodiesel No. 2-D and exceeds that for No. 1-D, 4.1 and 2.4 mm^2/s, respectively, the viscosity of biodiesel must be taken into account when blending. It should also be noted that biodiesel boiling points are those of the esters of the individual lipids, which are in the approximate range of 320°C to 350°C. ASTM PS 121 (D 6751) was developed with the philosophy that if the B100 met PS 121 (D 6751) and the petroleum diesel met ASTM D 975 then the two can be blended together up to 20% biodiesel with levels higher than B20 subject to engine manufacture experience. This is similar to how blends of No. 1-D and No. 2-D are handled commercially. There is no separate set of properties identified in PS 121 for the biodiesel blends. Hence the development of a commercial item description for B20, which specifies some blend properties, was devised. [1]

Table 3. Fuel Inspection Properties [9]

Property	ASTM Method	B100*	DF-2			LSRD-4			JP-8		
			DF-2	B20	B30	LSRD-4	B20	B30	JP-8	B20	B30
Density, 15°C, kg/L	D 1298	0.8839	0.8494	0.8597	0.8603	0.8509	0.8566	0.8613	0.8064	0.8218	0.8310
Flash Point, °C	D 93	163.5	75.5	79.5	82.5	74	76	78.5	47.5	50	51.5
Cloud Point, °C	D 2500	-4.2	-9.4	-8.7	-8.4	-17.9	-16.1	-14.9	-55.5	-27.2	-22.1
Pour Point, °C	D 97	-9	-18	-27	-30	-27	-24	-30	-69	-45	-33
Freezing Point, °C	D 2386	+5.9	-5.9	-5.7	-5.2	-14.8	-12.7	-11.3	-53.2	-16.9	-11.1
Viscosity, 40°C, mm^2/s	D 445	4.00	2.90	3.11	3.20	2.48	2.77	2.92	1.30	1.62	1.87
TAN, mg KOH/g	D 664	0.15	0.05	0.09	0.12	0.01	0.07	0.11	0.01	0.04	0.09
Total Water, ppm	D 4928	639	104	147	206	46	122	167	44	114	157
HFRR, mm	ISO	0.270	0.450	0.135	0.135	0.195	0.190	0.175	0.685	0.260	0.190
SLBOCLE, g	D 6078	6000	4700	5100	5100	3900	4175	6000	1850	4950	6000
BOCLE, mm	D 5001	0.68	0.58	0.54	0.58	0.57	0.55	0.58	0.55	0.54	0.61
Distillation, °C	D 86										
Initial		317	205	205	204	188	193	193	159	163	161
10% off		334	237	244	247	215	221	226	180	183	186
50% off		337	271	287	296	259	277	290	204	215	228
90% off		348	321	335	338	317	332	335	236	334	338
95% off		347	340	344	345	332	339	341	246	339	340
Final		347	357	352	350	344	346	346	260	343	343
Residue, wt%		4.3	1.5	1.3	2.1	1.9	1.3	1.3	1.0	1.2	1.2
*Methyl soyate											

The effect of lowering the cloud point by adding LSRF-4, DF-2 and JP-8 to the biodiesel is approximately linear for the diesel fuels but dramatically non-linear for JP-8 as plotted in Figure 1.

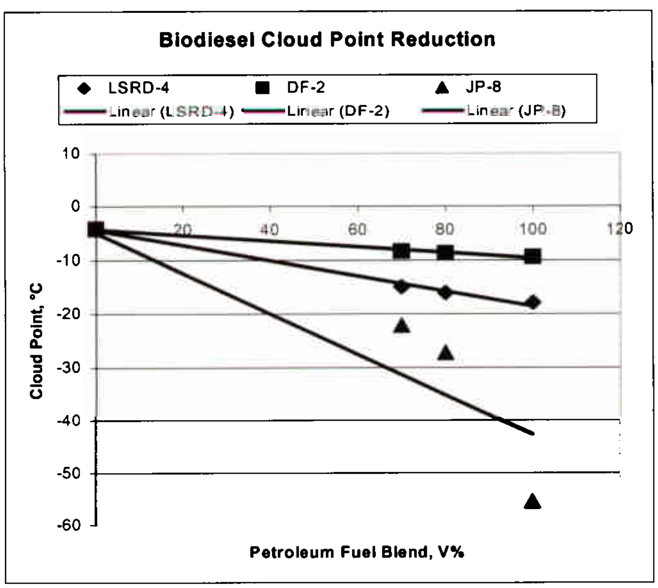

Figure 1. Cloud Point Reduction For Biodiesel

This may impact the ability to predict resultant cloud point of B20 when using diesel fuel oil grade 1-D for use in regions during very cold temperature conditions.

The fuel insolubles, acid number and viscosity of biodiesel and biodiesel blends can increase during storage. The rate of increase is a function of time and temperature w/rapid increases at elevated temperature.

Water solubility in biodiesel and biodiesel fuel blends stored under ASTM D 4625 43°C vented-conditions were observed to increase with time as demonstrated in Figure 2.

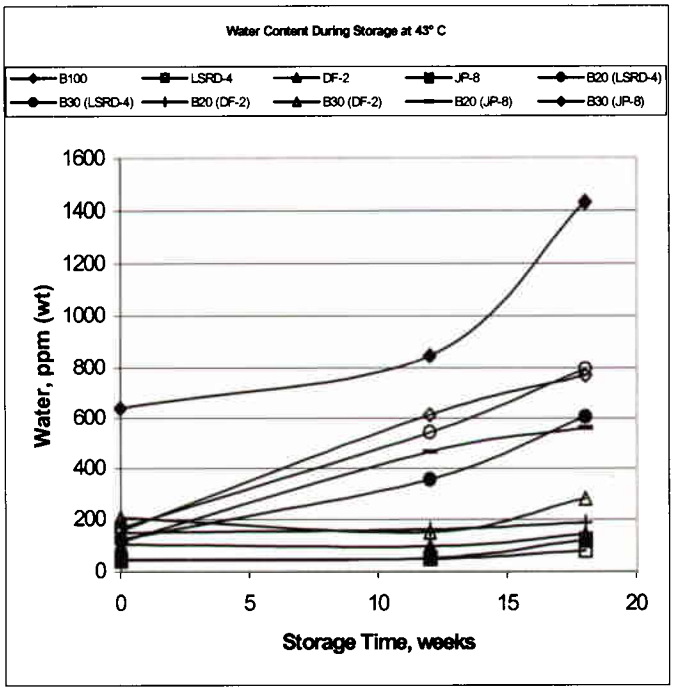

Figure 2. Water Adsorption in Fuel Stored Vented at 43°C (ASTM D 4625)

Looking more closely at the acid number increases in the fuel with time also suggested that the water absorption might be related to acid number increases. The acid number was plotted against total measured water in Figure 3 for the ten fuel samples after storage for 18-weeks, which confirms an increase in acid number with attendant increase in total dissolved water.

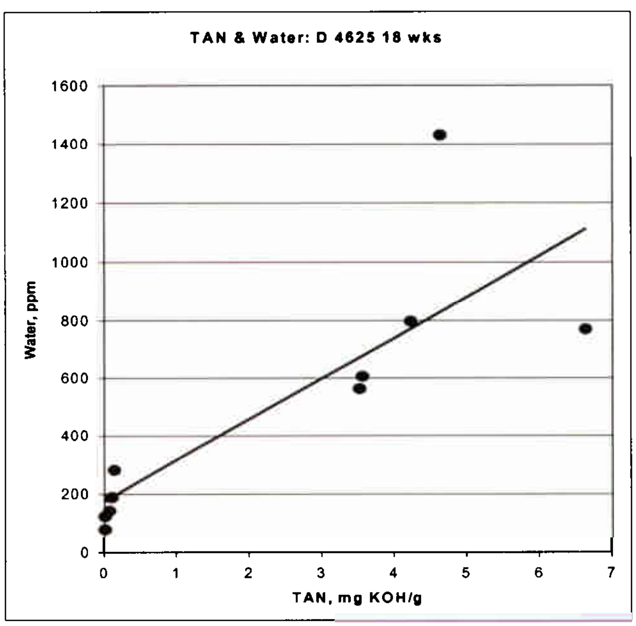

Figure 3. Absorbed Water Versus Total Acid Number After Vented Storage at 43°C

JP-8 jet fuel and low sulfur diesel fuel (LSDF) inherently tend to have lower lubricity than petroleum high sulfur diesel fuel. The lubricity property of diesel fuel is a measure of how well chemical components in the fuel prevent wear on contacting metal surfaces. High lubricity is important for rotary and distributor type fuel injection pumps in which the moving parts are lubricated by the fuel itself, not by the engine oil. [11] Laboratory tests have shown a 20 percent blend of biodiesel brings the lubricity level of neat JP-8 and LSDF to acceptable levels. Laboratory tests have also shown engine exhaust particulate matter is reduced when 20 percent biodiesel is blended with diesel fuel.

Biodiesel, especially if produced from highly unsaturated oils, oxidizes more rapidly than diesel fuel. The results of experiments to track the chemical and physical changes that occur in biodiesel as it oxidizes have been reported. These results show the impact of time, oxygen flow rate, temperature, metals, and feedstock type on the rate of oxidation. Blending with diesel fuel and the addition of antioxidants are explored also. The data indicate that without antioxidants, biodiesel will oxidize very quickly at temperatures typical of diesel engines. This oxidation results in increases in peroxide value, acid value, and viscosity. While the peroxide value generally reached a plateau of about 350 meq/kg ester, the acid value and viscosity increase monotonically as oxidation proceeds. [12]

A literature evaluation of potential oxidative and thermal stability test methods for biodiesel and biodiesel blends with petrodiesel, as well as the known effects of stability-related issues on performance in the field, has been completed. The advantages and disadvantages of the selected potential test methods were compiled to form a basis for further consideration and rating of the test methods. The literature search and rankings were peer reviewed by experts in the oleochemical, petroleum diesel, and diesel engine manufacturing fields. Based on the peer-reviewed rankings, limited bench-scale testing was performed on selected test methods and various modifications to determine their applicability to biodiesel and biodiesel blends. For various reasons, none of the methods (as written) appeared to be reliable indicators of performance with biodiesel or its blends. [6]

Modifications of ASTM D 2274 (Accelerated Method), the ASTM 150°C high-temperature stability test (commonly referred to as the duPont F-21 or Thermal Stability), and ASTM D 4625 (43°C Long Term Storage) test methods were identified that may enhance their ability to accurately and reliably predict the performance of biodiesel and biodiesel blends. A working group was formed within the biodiesel task force of ASTM to further evaluate biodiesel stability testing methods. The information developed from this effort will be summarized in an appendix of the ASTM standard for biodiesel (currently a provisional standard), which is similar to that contained in the ASTM petroleum diesel fuel standard, ASTM D 975. [6]

The effect of antioxidant butyl hydroxy toluene (BHT) on the reduction of B100 insolubles, acid number, and viscosity increase are shown in Fig. 4.

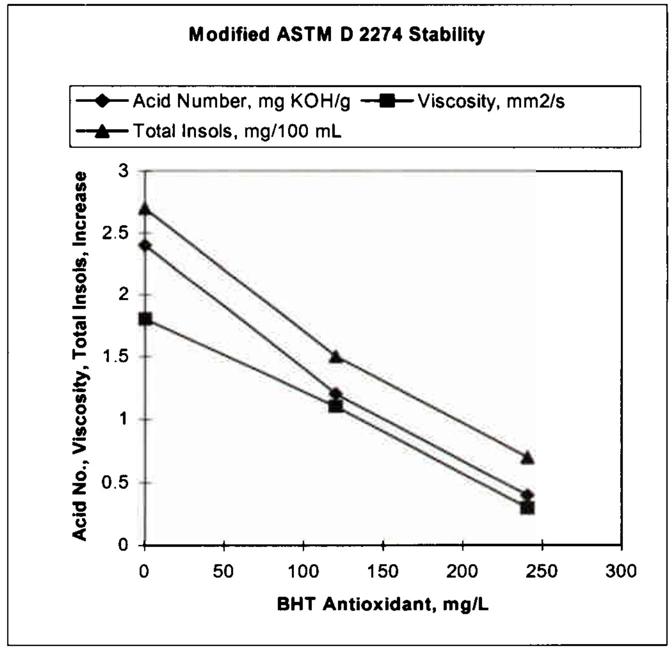

Figure 4. Insolubles, Viscosity and Acid Number Increases Reduced By Antioxidant at 80°C[6]

Goodman has indicated that biodiesel is not approved for use in tactical vehicles because of concerns regarding its long-term stability. [14] However, it may be suitable for use in administrative (commercial) vehicles where long-term storage is not an issue. Before deciding to use biodiesel, DOD fleet managers should ensure that:

- Separate tankage (not used by tactical vehicles) is available for biodiesel.
- The biodiesel being used meets the ASTM D 6751 specification for Biodiesel Fuel (B100) Blend Stock for Distillate Fuels.
- The impact of biodiesel use on warranty coverage, which varies by vehicle/engine manufacturers, has been checked. (Major engine manufacturers have all issued statements regarding the use of biodiesel as it pertains to their warranty coverage. To obtain a copy of any of these statements, contact the National Biodiesel Board).
- The higher cloud and pour points of biodiesel, which affect cold weather performance, are taken into account. (Cold flow properties of B20 are generally 1°C to 3°C higher than the neat diesel if it has a cloud point lower than about –10°C).

Although biodiesel B20 costs more than petroleum diesel, it can be used in unmodified diesel engines. Therefore, it represents an opportunity to meet a portion of DOD's AFV acquisition requirements at a lower overall cost. The Department of Energy has issued guidance "Biodiesel Fuel Use Credit Interim Final Rule" that provides details regarding how to use biodiesel to meet AFV acquisition requirements. [15] Goodman urged DOD to evaluate the use of biodiesel as an option for meeting a portion of your AFV requirements. Additional information on biodiesel is available from:

- National Biodiesel Board at (800) 841-5849 or www.biodiesel.org
- U.S. Department of Agriculture, Ms. Sharon Holcombe at (202) 720-3820
- U.S. Army Tank-automotive and Armaments Command (TACOM), Mr. Luis Villahermosa at (586) 574-4207
- U.S. Department of Energy at http://www.afdc.nrel.gov/

ALTERNATIVE FUELS AND BIODIESEL EXHAUST EMISSIONS

Alternative fuels are substantially non-petroleum and yield energy security and environmental benefits. DOE currently recognizes the following as alternative fuels: biodiesel, electric fuel, solar fuel, hydrogen, p-series, LNG, CNG, LPG, methanol (including M85) and ethanol (including E85). DOE can expand this list when new fuels are developed and approved as meeting this definition. EPACT recognizes the following as alternative fuels: methanol, denatured ethanol and other alcohols, mixtures containing 85 percent alcohol and 15 percent gasoline, natural gas, liquefied petroleum gas, hydrogen, coal-derived liquid fuels, electric (including solar), and fuels derived from biological materials. [16]

B20 biodiesel fuel blend is a cleaner-burning diesel fuel that includes natural, renewable sources such as transesterified vegetable oils. Just like petroleum diesel, B20 biodiesel fuel blend operates in compression-ignition engines. Essentially no engine modifications are required, and B20 biodiesel maintains the payload capacity and range of diesel. The use of B20 biodiesel blends in a conventional diesel engine results in substantial reduction of unburned hydrocarbons, carbon monoxide, and particulate matter. Emissions of nitrogen oxides (NO_x) are either slightly reduced or slightly increased depending on the duty cycle and testing methods. The use of biodiesel decreases the solid carbon fraction of particulate matter (since the oxygen in biodiesel enables more complete combustion to CO_2), eliminates the sulfate fraction (as there is no sulfur in the fuel), while the soluble, or hydrocarbon, fraction stays the same or is increased. Therefore, biodiesel works well with new technologies such as catalysts (which reduces the soluble fraction of diesel particulate but not the solid carbon fraction), particulate traps, and exhaust gas recirculation (potentially longer engine life due to less carbon and higher lubricity). [17]

Tyson has edited a comprehensive summary of relevant biodiesel and biodiesel-related research, development, demonstration, and commercialization projects completed and/or started in the United States between 1992 and 1997. [18]

Schmidt, et al., has concluded that biodiesel's particulate reducing effect can be attributed to two factors: its displacement of aromatics and shorter chain paraffin hydrocarbons and its oxygen content. [19]

Durbin, et al., reported four heavy-duty diesel trucks on chassis dynometer tests showed that biodiesel, 20 percent biodiesel blend, and a synthetic diesel produced generally lower THC and CO emissions than California diesel. [20] The NO_x emissions were comparable over most of the fuel/vehicle combinations, with slightly higher NO_x, emissions found for the two noncatalyst vehicles on 100 percent biodiesel.

Biodiesel can be used in existing diesel engines without modification; however, property changes associated with the differences in chemical structure between biodiesel and petroleum-based diesel fuel may change the engine's injection timing. [20] These injection timing changes can change the exhaust and performance from the optimized settings chosen by the engine manufacturer. The results of measurements of the speed of sound and the isentropic bulk modulus for methyl and ethyl esters of fatty acids from soybean oil and compares them with No. 1 and No. 2 diesel fuel. Data are presented at 21 ±1°C and for pressures from atmospheric to 34.74 MPa. The results indicate that the speed of sound and bulk modulus of the monoesters of soybean oil are higher than those for diesel fuel and these can cause changes in the fuel injection timing of diesel engines. This may explain equal or slightly increased NO_x values associated with biodiesel and biodiesel blends. [21]

Schröder has also reported similar NOx results for exhaust emissions from rape seed oil methylester. [22] In Germany 100,000 tons of biodiesel (rapeseed oil methylester, RME) were produced in 1998; 200,000 tons are expected in 2000.

ARMY FLEET TESTS USING BIODIESEL

Frame [9] developed a comprehensive Army Biodiesel Fuel Evaluation Program plan that was to involve a three-phase approach: laboratory testing, engine dynamometer testing, and field validation. However, funding was only provided for the first phase.

Two modest, less thorough, Army tests are discussed in Reference 1. In the larger test, there were no initial checks on the vehicles at the start of their testing, no new fuel filters installed when they began to "test" the JP-8 with 20 percent methyl soyate, no fuel sample property analyses performed on the resultant blends, no control vehicles, and interesting, but questionable use of the JP-8/methyl soyate blend instead of using B20. A biodiesel B20 fuel evaluation was performed on various U.S. Army tactical wheeled vehicles from March 1994 through March 1995 at the U.S. Army Yuma Proving Ground (YPG). [23] Testing was conducted to compare vehicle system performance when the vehicles were operated with an 80/20 percent JP-8/biodiesel fuel blend instead of neat (or 100 percent) JP-8 fuel. The vehicle types under test included the Commercial Utility Cargo Vehicle (CUCV), High Mobility Multipurpose Wheeled Vehicle (HMMWV), M939A2 Series of 5-Ton Truck, Heavy Expanded Mobility Tactical Truck (HEMTT), and the M915A2 Truck-Tractor.

After mixing, a sample of the JP-8/biodiesel blend was drawn and submitted for chemical analysis. This was done to assure proper pre-operations of the neat fuels were present. The method developed to determine the content of the blend involved using a Nicolet 510p infrared spectrophotometer. The analysis technique revolved around the biodiesel having an absorbency band at 1750 cm^{-1}, which is very weak in neat JP-8. This band was used as the qualification band with four standards ranging from 0 to 30 percent in 10 percent intervals being measured. A calibration curve was developed based on the absorbency measurements of the standards. The results of the analysis revealed the batches under test ranged from a minimum of 18.6 percent to a maximum of 23.0 percent neat biodiesel content. [23]

A second fleet test of much smaller dimension than the Yuma demonstration was successfully accomplished as a trial of B20 at Fort McCoy during 1993-1994. [24] Of ten buses at Fort McCoy, two were operated on B20, without difficulty.

The fleet test using B20 biodiesel at USDA included one surplused military vehicle, but does not have control vehicles operating on petrodiesel for comparison. [25]

ORIGINAL EQUIPMENT MANUFACTURER'S POSITION ON BIODIESEL

Some of the Original Equipment Manufacturer's (OEM) allowances for the use of biodiesel fuel blends were reviewed. Many of the statements may have been formulated before the ASTM PS 121 (ASTM D 6751) for biodiesel was established and are subject to change, thus their validity should be verified before accepting this information at face value. After considering the information, the TACOM draft Purchase Description (PD) for biodiesel B20 (having two grades, B20 LS 1-D and B20 LS 2-D) may need to state that before use in equipment, contact equipment manufacturer for guidance. This is based on some of the current original equipment manufacturer statements or considerations, which included:

- Manufacturer may recommend oil change period may be reduced.
- Manufacturer may require that biodiesel B20 grade No. 1 distillation limits be the same as those for Low sulfur Diesel grade No. 1-D, which is impossible due to high boiling point of biodiesel.
- Manufacturer may require that the biodiesel be manufactured from virgin soybean oil or rapeseed oil.
- In field trials, the following injection equipment and engine problems have been identified as being caused by these fuel characteristics (solvency of fatty acid methyl esters, oxidation derived acids and insolubles): fuel leakage, filter plugging lacquer formation in hot areas. The incidence of these effects may be increased when engine is in irregular use, in applications such as stand-by generator units, automatic plant and seasonally used vehicles. [26]

- Cellulosic filters may be compromised due to solubility of resin and binders in biodiesel component of biodiesel B20, allowing filter migration. Glass fiber based fuel filters manufactured without the use of binders probably are not affected.
- One manufacturer's suggested limit of 15 mg/L for ASTM D 2274 accelerated stability can be met but the mixed cellulosic ester filter used in the test method is not compatible with soy methyl ester (which can actually dissolve the filter) and should be replaced with glass fiber (GF/F). [6]

Since the adoption of the ASTM PS 121 and currently ASTM D 6751, it is expected that positions related to B20 use may reflect the specification, especially when it becomes a full specification.

"Diesel Fuel Injection Equipment (FIE) manufacturers fully support the development of alternative sources of fuel for compression ignition engines. In Europe and in the United States, fuel resources such as Rape Methyl Ester (RME) and Soybean Methyl Ester (SOME), collectively known as Fatty Acid Methyl Esters (FAME) are being used as alternatives and extenders for mineral oil derived fuels. The FIE manufacturers are aware of issues peculiar to fatty acid methyl ester fuels and are active in the generation of Standards for these fuels to protect the end-users of their products from potential premature failure. Biodiesels must conform to such Standards to be of acceptable quality, just as mineral oils do at present. To date, experience in Europe has been mainly associated with the methyl esters of Rapeseed oil and in the US with Soybean derived fuels. Whether or not the service experience with these fuels will apply/extend to all fatty acid methyl esters (including such as Tallow ME and Used Frying Oil ME) has yet to be determined. FAMEs tested so far appear to have good lubricity and cetane numbers. As currently manufactured, these fuels are less stable than mineral oil derived fuels. FAME fuels readily "bio-degrade" in the event of accidental spillage or leakage - this is claimed as a marketing advantage-The degradation propensity is, however, of major concern to the FIE manufacturers as the products of this natural process can be potentially harmful to the fuel system." [26]

Paul Henderson has provided a letter to express support for Kansas House Concurrent Resolution No. 5069, which encourages the use of biodiesel in low blend levels in the State of Kansas. [26] Henderson states "Through cooperation with the National Biodiesel Board, we have tested biodiesel at Stanadyne and results indicate that the inclusion of 2 percent biodiesel into any conventional diesel fuel will be sufficient to address the lubricity concerns that we have with these existing diesel fuels. From our standpoint, inclusion of a 2 percent biodiesel fuel blend is desirable for two reasons. First, it would eliminate the inherent variability associated with the use of other additives and whether sufficient additive was used to make the fuel fully lubricious. Second, we consider biodiesel a fuel or a fuel component--not an additive. It is possible to burn pure biodiesel in conventional diesel engines. Thus, if more biodiesel is added than required to increase lubricity, there will not be the adverse consequences that might be seen if other lubricity additives are dosed at too high a level." [27]

Technically, the positive attributes of the biodiesel B2 (2 percent biodiesel blend in petrodiesel) supported by Paul Henderson may also overcome many of the negative attributes of biodiesel B20. Note that biodiesel B2 does not enjoy EPACT credits.

A large variety of biodiesel B100 samples have been shown to be effective in reducing the HFRR (60°C) of a JP-8 fuel from 735 micrometers to less then 480 micrometers at or below concentrations of 1 percent. [28] Biodiesel, as a lubricity additive, may have a high value place to play in premium diesel.

PREMIUM DIESEL

The National Conference on Weights and Measures (NCWM) in the U.S. are in the process of defining a higher quality diesel fuel for modern diesel engines, which has been termed Premium Diesel. [29] Premium Diesel will be defined as a diesel, which has at least two of five fuel-performance properties superior to the U.S. standard diesel fuel (ASTM D 975). The five premium diesel properties are energy content, cetane number, thermal stability fuel injector cleanliness, and low temperature operability. [30]

Lubricity was also considered as a premium diesel criterion. Poor lubricity fuels can lead to accelerated wear in fuel lubricated, rotary type, fuel injection pumps. However, the two standard test methods for lubricity under consideration, the Scuffing Load Ball on Cylinder Lubricity Evaluator (SLBOCLE) and the High Frequency Reciprocating Rig (HFRR) have large repeatability and reproducibility limits making enforcement of a limit difficult. They are also less sensitive to additives than desired.

At least one diesel fuel company is distributing preblended biodiesel in petroleum fuel. Koch introduced its soy-based fuels in Iowa, Kansas, Nebraska, Minnesota, North Dakota and South Dakota. The company said the soy-based fuels, which are the nation's first pre-mixed, precision-blended soybean oil fuels, were primarily available to farm customers in those areas. However, the company decided to add more distribution locations in South Dakota and expand its offerings into Iowa. Koch said it was the first company to create a biodiesel "already blended at the terminal, saving diesel users from the guesswork and inconvenience of mixing." The company said the soy-based fuels provide better fuel economy and more power than conventional diesel fuel. In addition, Koch said its biodiesel fuels offer improved lubricity. [31] In 8 June 2000, "Koch Performance Fuels today announced the expansion of its high-quality, environmentally friendly fuel, U.S. Soy 50 Field Diesel™, into Michigan."

CHARACTERIZATION DATA TO SUPPORT CID FOR DIESEL FUEL, BIODIESEL BLEND (B20)

A project to develop data to characterize selected biodiesel samples and biodiesel (B20) blends made with diesel fuels was used to support a CID A-A-59693 specification for Biodiesel Blend B20 Diesel Fuel. [33] The project was initiated to evaluate a variety of biodiesels (identified in a market survey, TARDEC Technical Report No. 13801) from different manufacturers, sources, and types (unused versus used). [34] Ten (10) companies (eight (8) manufacturers and two (2) suppliers) were contacted to participate in this study. Nine of the ten companies agreed to participate and provided a total of 15 samples of B100 for testing. The feedstocks used in the manufacture of the biodiesel samples provided were as follows: a) unused soybean cooking oil, b) used soybean cooking oil, c) unused vegetable oil, d) used vegetable oil, e) unused canola, f) unused cottonseed, and g) yellow grease. Data are summarized in Appendix A.

Two blends of B20 were made from each sample of B100 received. One sample was blended with low sulfur No. 1 diesel fuel (LS-1D) and the second sample was blended with low sulfur No. 2 diesel fuel (LS-2D). Two B100 samples (sample numbers 14 and 15 in Table 5) were not used in blends due to the small quantity provided for testing. Therefore, fifteen (15) B100 samples and twenty-six (26) B20 samples were analyzed in addition to the two low sulfur diesel fuels. Various chemical and physical properties were determined on all of the samples to ensure compliance with B20 and B100 specification requirements.

The analysis of the test results for the neat biodiesel and biodiesel blends showed that an acceptable fuel for non-tactical vehicle (NTV) consumption can be manufactured. There are some properties that particular attention needs to be addressed. Cleanliness/particulate content, acid number, and low temperature properties are properties that biodiesel seems to affect the most – even in blends. These properties must be monitored and controlled to ensure that the user is not left to face problems later.

The manufacturers of biodiesel and biodiesel blends need to be aware of these issues/concerns since they can control these properties during the manufacturing/blending process and ensure that the delivery of the product is done from clean tankage to avoid contamination during delivery.

The user must recognize and be aware of these problems, particularly the cloud point effects, to avoid the possibility of across-season usage of fuel. Since biodiesel increased the cloud point of the base fuel between 2° to 20 °C in this study, it highlights the importance of avoiding the use of fuel procured for a warmer season during winter. Monitoring the acid number will help assess whether the product is deteriorating before the rancid smell, typical of deteriorated biodiesel, can be detected in stored products.

A select number of tests were conducted on the samples received and the blends. Table 4 provides a list of the performance tests conducted and reported in Section 4. Experimental Results.

Table 4. Test Performed for B100 and B20 Diesel Fuel.

Performance Test	Method	Tests Performed	
		B100	B20/DF-1/ DF-2
Appearance	ASTM D 4176	X	X
Kinematic Viscosity @ 40°C	ASTM D 445	X	X
Flash Point, °C	ASTM D 93		X
Total Acid Number	ASTM D 664	X	X
Particulate Content	ASTM D 6217		X
Water Content	ASTM D 4298	X	X
Distillation	ASTM D 86		X
MCRT	ASTM D 4530	X	X
Cloud Point	ASTM D 5773	X	X
Calc. iodine Value		X	
Oxidation Stability	ASTM D 6186 modified	X	

To determine the effects of different feedstocks, both unused and used, as well as the type of feedstock, various chemical and physical (C&P) properties, as shown in Table 4, were evaluated. The C&P properties were reviewed to determine the impact to base fuel and whether the samples can comply with B20 and B100 specification requirements. Two blends of B20 (i.e., a fuel blend consisting of 20% volume biodiesel and 80% volume petroleum-derived diesel) were made from each sample of B100 received. One sample was blended with low sulfur No. 1 diesel fuel (LS 1-D) and the second sample was blended with low sulfur No. 2 diesel fuel (LS 2-D). Two B100 samples (sample numbers 14 and 15 in Table 5) were not used in blends due to the small quantity provided for testing. Therefore, fifteen (15) B100 samples and twenty-six (26) B20 samples were analyzed in addition to the two low sulfur diesel fuels. Table 5 lists the company code, type feedstock, biodiesel sample number, assigned laboratory code, and the laboratory codes of the B20 blends made with LS 1-D and LS 2-D. The B20 blends laboratory codes maintained the same middle numbers as the B100 assigned laboratory codes and was preceded by either D1 or D2 depending on the base fuel used in the blend and ended with "20" to reflect a 20% blend. This allowed quick determination on which B100 and base fuel were used in the blends.

Table 5. Biodiesel Sample Identification.

Company Code	Type Feedstock	Biodiesel Sample Number	Laboratory Code FLTT Identification No.	B20 Blend with LS 1-D	B20 Blend with LS 2-D
A	Unused soybean from USADA	Sample 1	FL-11235-00	D1-11235-20	D2-11235-20
B	Unused soybean	Sample 2	FL-11237-00	D1-11237-20	D2-11237-20
C	Unused Soybean	Sample 3	FL-11250-00	D1-11250-20	D2-11250-20
D	Unused soybean	Sample 4	FL-11257-00	D1-11257-20	D2-11257-20
E	Unused soybean	Sample 5	FL-11262-00	D1-11262-20	D2-11262-20
F	Used Cooking Oil	Sample 6	FL-11271-00	D1-11271-20	D2-11271-20
G	Used Cooking Oil	Sample 7	FL-11272-00	D1-11272-20	D2-11272-20
H	Used soybean cooking oil	Sample 8	FL-11273-00	D1-11273-20	D2-11273-20
A	Unused soybean	Sample 9	FL-11274-00	D1-11274-20	D2-11274-20

Company Code	Type Feedstock	Biodiesel Sample Number	Laboratory Code FLTT Identification No.	B20 Blend with LS 1-D	B20 Blend with LS 2-D
I	Unused canola	Sample 10	FL-11316-01	D1-11316-20	D2-11316-20
I	Unused cottonseed	Sample 11	FL-11317-01	D1-11317-20	D2-11317-20
I	Yellow grease	Sample 12	FL-11318-01	D1-11318-20	D2-11318-20
I	Unused soybean	Sample 13	FL-11341-01	D1-11341-20	D2-11341-20
A	Unused vegetable oil blend	Sample 14	FL-11291-00		
A	Used vegetable oil blend	Sample 15	FL-11292-00		

In order to determine the effect of biodiesel feedstock type on B100 and B20 fuel, the following properties were determined for both B100 and B20 samples: Kinematic viscosity @ 40°C, total water content, cloud point, pour point, freezing point, visual appearance, total acid number (TAN), micro carbon residue test (MCRT), density, and particulate content. For baseline purposes the low sulfur diesel fuels were also tested. This data is tabulated in Appendix A.

Kinematic Viscosity @ 40°C: The data shows that the viscosity of biodiesel is unrelated to whether the product is used or unused. However, the original feedstock properties will have a greater impact on viscosity, demonstrated by the range of viscosity of the biodiesel samples using the same type of feedstock.

Flash Point: *In general, the blending of biodiesel and petroleum fuel gave an increased flash point or remained the same for B20 as compared to the diesel fuels themselves.*

Cloud Point: *The great variance and increased cloud point when blending biodiesel, particularly when using LS 1-D fuel, highlights the importance of proper monitoring of the finished fuel properties to ensure that the specification limits are met.*

Total Acid Number (TAN): *The acid numbers are believed to be a reflection of the manufacturing process and not a direct measure of biodiesel feedstock type. The B20 specification, A-A-59693, requires the TAN in B20 not to exceed 0.25 mg KOH/g.*

The LS 1-D and LS 2-D fuels used for blending had the same total acid number of 0.0014 mgKOH/g. All B20 blends had a higher total acid number as compared to the base diesel fuels. Biodiesel Sample 4 had the lowest acid number and exhibited the smallest change in acid number, 0.0147mgKOH/g. The B20 samples blended with LS 2-D tended to have a higher total acid number than the B20 samples blended with LS 1-D. Biodiesel Sample 12 was the only sample to fail to meet the specification requirement for B20 of 0.25mgKOH/g.

Total Water Content: *Total acid number of a biodiesel B100 tends to correlate to total water content determined by ASTM D 4928. The data in Fig. 5 demonstrates a fair correlation with an R-squared value of 0.63 between TAN and total water content. In previous work [1] a relationship between TAN and water was discussed which is supported by this work.*

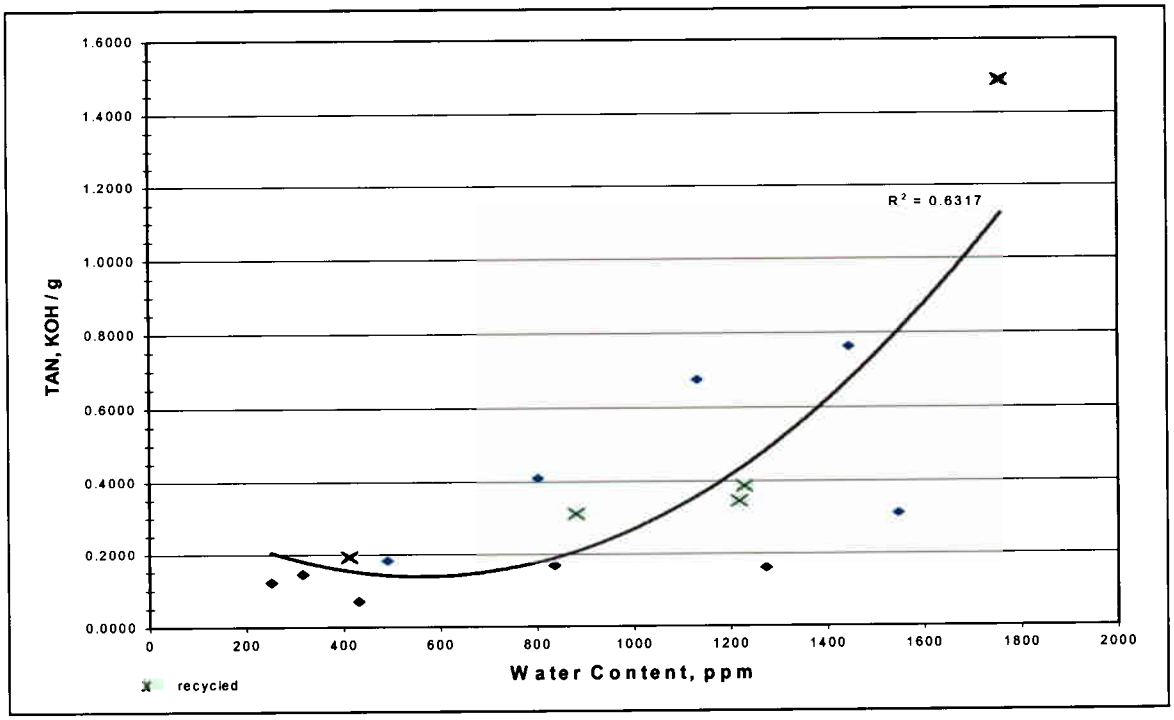

Figure 5. Correlation of TAN and Total Water in Biodiesel.

None of the biodiesel samples had any free water, as indicated by the visual inspection of the samples.

Particulate Content: High particulate content may result in vehicle fuel filters being prematurely plugged resulting in a higher replacement rate. Depending on the type of particulates, it can also wear fuel pumps and injectors prematurely. The particulate content of a fuel can be a result of manufacturing, transport, delivery, and storage of the product.

Biodiesel has a better solvency than diesel fuel, which can help loosen sediment in fuel tanks and result in increased particulate content. Particulate content in biodiesel B20 blends was determined using ASTM D 6217 with glass filter fibers (GF/F). Some of the samples were high in particulate content but within maximum limits indicated in A-A-59693, except for sample No. 2, which failed. The B20 particulate requirement in A-A-59693 is restricted to 10 mg/L, maximum. The biodiesel Sample 12 had a greater than expected variance between the two base fuels. An explanation for this variance was not explored at this time. However, it is speculated that the base fuel LS 1-D has a better solvency than LS 2-D and could potentially dissolve insolubles resulting in a lower particulate content value.

The increase of particulates from the petroleum stock to the B20 blends appears to be slightly higher for LS 2-D, in general.

Micro Carbon Residue Test (MCRT): *The maximum specification limit for ASTM D 6751 was 0.05 mass percent. Five of the fifteen B100 samples failed to meet specification requirements.*

The petroleum diesel fuels were tested via the same MCRT method and gave results of zero (<0.0001) mass percent. The maximum specification requirement for B20 was 0.05 mass percent. The results for all samples fell below the biodiesel B20 blend specification requirement; however, the blend's carbon residue corresponds to the B100 carbon residue values.

It appears that the type of feedstock used to manufacture the biodiesel impacts the micro carbon residue results. The biodiesel made from soybean showed the least amount of carbon residue in the B100 and the B20 samples.

Distillation (T90): ASTM D 975 is the commercial specification for LS 1-D and LS 2-D and it lists T90 maximum limits of 288°C and 338°C, respectively. The LS 1-D used for blending had a distillation (T90) temperature of 250°C. The T90 of the B20 samples blended with LS 1-D averaged 333.5°C and were all significantly above the 288°C limit. Therefore, none of the B20/LS 1-D samples met the LS 1-D fuel specification requirement for T90.

The LS 2-D T90 specification range is 282-338°C. The base fuel used as the L2SD had a T90 of 321°C. The B20 samples blended with LS 2-D averaged a T90 of 338.4°C. The average result for the used B20/LS 2-D was 335°C while for the unused B20/LS 2-D samples the average was 339°C, which is just above the ASTM D 975 limit.

All biodiesels raised the T90 distillation point of the finished biodiesel B20 blends. The increase was not associated with any one type of biodiesel feedstock. This increase is expected due to the higher boiling point of neat biodiesel.

A gas chromatographic method was developed to quantitate biodiesel in B20 biodiesel blends using a modification of ASTM D 2887. [33] This method takes advantage of the high boiling point property of biodiesel.

Other Properties

Several other properties not listed in the commercial item description (CID) A-A-59693 for B20, ASTM D 6751, or ASTM D 975 were evaluated during this project. These properties were as follows: Pour point, freezing point, and density. Additionally, pressure differential scanning calorimetry (PDSC) was performed on the B100 samples and selected B20 samples made from the most reactive biodiesel. These data aid in the characterization of biodiesel and biodiesel blends.

Pour point: Biodiesels manufactured from unused feedstocks generally have lower pour points than biodiesels manufactured from used feedstocks. The very low pour point value (-18°C) for Sample 5 was unusual and may suggest that this biodiesel contains pour point depressant.

Pure biodiesel (B100) does not have very low pour points. The figure also shows that biodiesel from unused feedstocks seem to have lower pour points than the used feedstocks. Since used feedstocks are initially designed for high temperature cooking, this is not unexpected since they generally will contain less of the very reactive unsaturation present in soybean oil feedstock.

LS 1-D blends also exhibit lower pour points than the LS 2-D blends. The base LS 1-D pour point was -62°C while the base LS 2-D fuel had a pour point of -28°C. The addition of the B100 to the petroleum fuels raised the pour points of the B20 blends as expected. However, potential low temperature problems are better determined by the use of cloud point.

Biodiesel Samples 1, 5, 10, and 9 exhibited the lowest pour points when blended with LS 1-D. The B20/LS 2-D blends have similar pour points with no significant difference between types of feedstock used. However, the addition of the biodiesel to the low sulfur diesel fuels showed a quite significant increase. The difference between the LS 1-D and B20/LS 1-D was –28.6°C while the difference was –8.2°C between LS 2-D and B20/LS 2-D. This is the same effect we observed with the cloud point discussed earlier.

Freezing Point On average, the products manufactured with used feedstocks (6.1°C) had higher freezing points than the unused feedstocks (3.3°C).

The freezing points of the low sulfur diesel fuels are greatly increased with the addition of the feedstocks to the formulations. LS 1-D, the winter diesel fuel, has a freezing point of –46.3°C while the summer fuel, LS 2-D, has a freezing point of –11.7°C. The effects of adding bio-based material to the petroleum fuels are more pronounced for LS 1-D. The B20/LS 1-D averaged 33.8°C higher than the diesel fuel itself, and the B20/LS 2-D samples averaged 2.5°C higher than LS 2-D. This effect is essentially the same as what was found with cloud point.

Visual Appearance: The samples were all inspected visually for debris and clarity in accordance with ASTM D 4176, Standard Test Method for Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedures). The requirement in A-A-59693 is for samples to be "visually free from undissolved water, sediment, and suspended matter." The test rates the samples on a pass or fail basis. Three of the fifteen B100 samples failed the visual appearance test. Samples 2 and 12 were cloudy and sample 5 contained particulate matter. The remaining twelve samples were clear and bright. All B20/LS 1-D and B20/LS 2-D blends were clear and bright. This method states that under good viewing conditions, the eye is able to detect 40 ppm water in the vortex formed during this test

Density: B100 samples manufactured from unused feedstocks generally have higher densities. Sample 13 has the highest density (0.894 kg/L).

The density of the B20 blends and the low sulfur petroleum fuels indicate that the addition of the biodiesel increases the density of the low sulfur petroleum fuels.

LS 2-D (0.8639kg/L) has a greater density than LS 1-D (0.8199kg/L). The addition of B100 to the petroleum fuels increased the density of all samples. B20/LS 1-D had the greatest increase in density with an average of 0.0131kg/L while B20/LS 2-D experienced a change of 0.0049kg/L. There are no major differences between the results for unused and used feedstocks.

Oxidation Stability: The oxidation stability of biodiesel (B100) was evaluated using first a modified ASTM D 5304, Standard Test Method for Assessing Distillate Fuel Storage Stability by Oxygen Overpressure. One test was completed at 146°C using the modified ASTM D 5304 procedure with a very soy biodiesel identified in all previous charts as Sample 6. The 2000 psia safety rupture disc burst during the first 10 minutes of the test. Other tests planned at 95°C could not be performed until the pressure reactor was repaired. The burnt fuel, shattered glass container, burst disc, pressure reactor, and other information were documented [35].

Due to time constraints and potential for other such damage to the test equipment, a modification was then used with ASTM D 6186, Standard Test Method for Oxidation Induction Time of Lubricating Oils by Pressure Differential Scanning Calorimetry (PDSC), as a test procedure for oxidation stability. The same biodiesel (Sample 6) was analyzed by PDSC, which gave an induction period of 3.0-minutes at 130°C and 4.4-minutes at 125°C (500 psig oxygen), accompanied by an audible pop at the induction time in both tests. This confirmed the potential of the PDSC as an oxidation stability method as suggested by Dunn [36]. We then continued the oxidation stability analysis of the remaining biodiesel samples using the 125°C temperature to ensure enough time to capture the induction period. The induction periods observed by the PDSC are shown in Fig. 6. The induction period (Fig. 6) for samples 8, 11, 12, and 13 reflect the time when the test was terminated and not necessarily an induction period for the samples.

During the evolution of using the PDSC test to evaluate biodiesel (B100), it was decided to run the samples up to a maximum of 60 minutes. Samples with times reported higher than 60 minutes were run before the decision top limit test time was agreed on.

It is interesting to note that 50% of the unused base stock samples (out of ten samples) had induction periods of less than 10 minutes while none of the used base stock samples had induction periods of less than 60 minutes. It is not possible to determine the reasons for this difference since we cannot validate whether any of the samples had anti-oxidation additives added or not. However, it is speculated that since the used samples were primarily used for cooking that the highly reactive compounds had already oxidized and removed from the biodiesel during the manufacturing process.

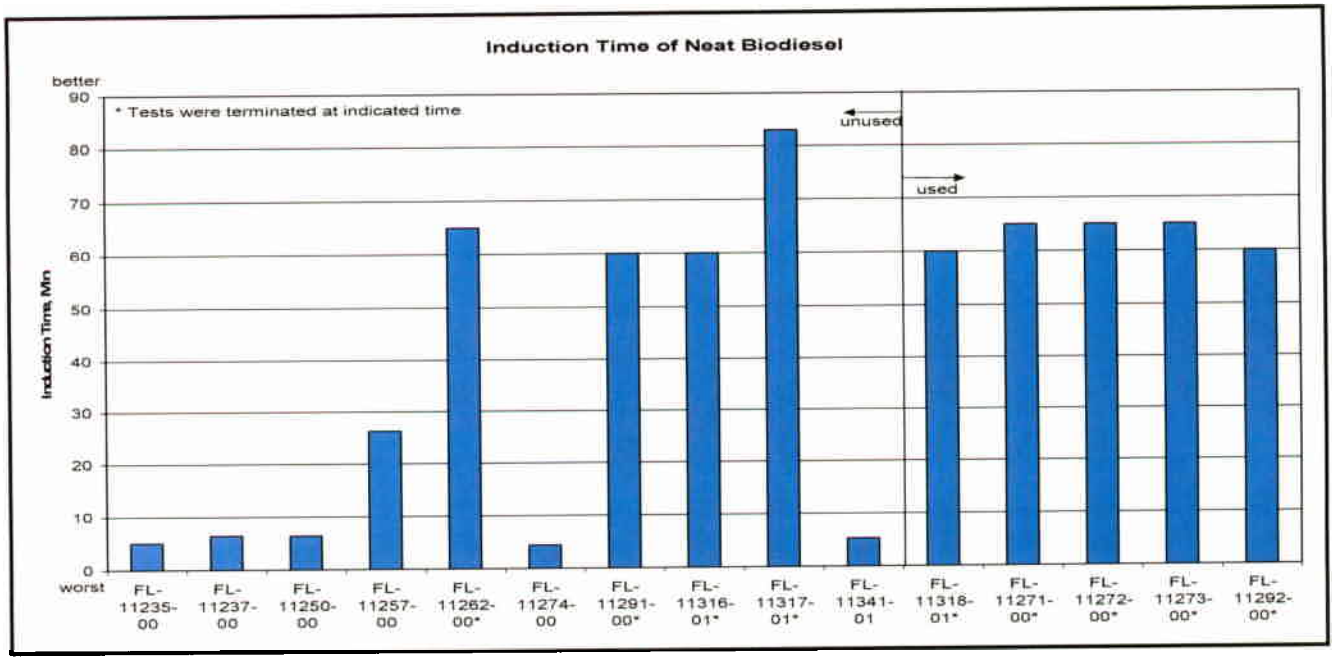

Figure 6. Induction Periods for B100 Fuel Samples at 125°C.

Particulate Contaminants: The Analysis of selected biodiesel B100 samples for particulate contamination by ASTM D 6217 and D 6426 is provided in Table 7. [37]

Table 7. Particulate Analysis for B100 Biodiesel Fuels

ASTM D 6217 - Particulate Contamination			ASTM D 6426 - Filterability of Distillate Fuel				
Sample ID	Nylon Filter mg/L	Glass Fiber Filter mg/L	Sample ID	mL	psig	F-QF**	Pass/Fail
FL-11237-00	33.6*	69.6*	FL-11237-00	250	15.1	42	Fail
FL-11262-00	56.4	56.4	FL-11262-00	62	15.0	10	Fail
FL-11271-00	64.2	64.8	FL-11271-00	22	15.0	04	Fail
FL-11274-00	1.2	1.4	FL-11274-00	300	5.2	83	Pass
				366	15.0	11	Pass (Fail at 366 mL)
FL-11316-00	8.0	8.0	FL-11316-00	200	15.1	33	Fail
FL-11318-00	6.2	6.2	FL-11318-00	21	15.0	04	Fail
* = Particles stuck to all glass surfaces, could not rinse off with toluene, actone, hexane, iso-octane, or methylene chloride.			**F-QF (300mL at P(F) = ((15psi - P(F)) / 15psi) x 50 + 50 F-QF (V(F) at 15psi) = V(F) / 6				

Both nylon (0.8 μm) and glass fiber filters (Whatman GF/F, 0.7 μm particle retension size) were used in ASTM D 6217 to determine particulate contamination. Note that the results agreed very well for both types of filters. These samples were also evaluated for filterability using ASTM D 6426 (with standard Fcells made with 5 µm nylon porous membrane material). The material in the biodiesel B100 appears to readily plug the Fcell filters.

In order to evaluate filterability with the vehicle fuel filter elements, filter elements were sent to EMCEE Electronics, Inc., (520 Cypress Ave.,Venice, FL 34292) for cutting into 25-mm diameter discs and placement in Fcells. The Fcell has an effective filtering area of 158.9 mm^2.

The actual vehicle fuel volume leading to failure (plugging) based on filter surface area and Fcell fuel volume to plug are summarized in Table 8.

Table 8. Prediction of Fuel Volume to Plug Filters Based On Fcell Test Data Using a B20 Fuel With D 6217 Particulate Level of 24 mg/L

Filter Identification	Filter Surface Area		Fuel Volume to Plug		
	in^2	cm^2	Fcell, D 6426, Liter	Vehicle Filter, Liter	Vehicle Filter, Gallon
(5 µm (Nylon in D 6426))*	(0.25)	(1.589)	(0.016)	(0.016, Standard Test)	(0.004, Standard Test)
3 µm commercial paper	400	2581	0.211	343	91
10 µm commercial paper	400	2581	0.376	611	162
FF1088	247	1594	1.635	1,640	434
FF898	128	826	N/A	N/A	N/A
FF863	326	2104	2.784	3,686	975
FF1005	215	1387	2.255	1,968	521
*Standard method results for comparison					

These data were generated using a B20 biodiesel fuel sample having an ASTM D 6217 micro-particulate level of 24 mg/L usind the standard 0.8 µm nylon pourous filter.

It is concluded that the ASTM D 6426 Fcell evaluations of biodiesel and biodiesel B20 blends using commercial fuel filter paper is a useful approach for correlating ASTM D 6217 microparticulate levels to vehicle fuel filter capacities or plugging limits.

It is recommended that D 6426 be modified to allow use of higher capacity filter media similar to that used in commercial vehicle diesel engine systems. It may be possible to use Whatman GF/F glass fiber filters, however, no data has been developed with Fcells employing this filter to provide comparative data. It is recommended that Fcells made with various types of laboratory grade filter media be evaluated with contaminated fuels. [37]

CHARACTERIZATION DATA TO SUPPORT BIODIESEL FILL AND GO SPECIFICATION (B2-B10)

A project to develop data to characterize selected biodiesel samples and biodiesel (B2-B10) blends made with diesel fuels was initiated to support recommendations for inclusion of biodiesel in the ASTM D 975 specification for diesel fuel. [38] Based on this data, it was apparent that inclusion of B5 biodiesel blend using both petroleum low sulfur diesel grades 1-D and 2-D could be transparent to the user if the biodiesel did not exceed 5 percent. For inclusion of B6-B20 biodiesel blends in ASTM D 975, it was recommended they be covered in a new annex to ASTM D 975. The B100 used for blending would be required to meet ASTM D 6751. The current CEN EN 590 specification for diesel fuel used in Europe includes the use of biodiesel in diesel fuel up to 5 percent.

The Engine Manufacturers Association (EMA) announced in March 2003 it has published a technical position statement on the use of biodiesel fuel in compression-ignition engines. [39]:

"The statement provides a factual assessment of biodiesel fuels and the potential effects of their use with current technology engines. The statement was prepared as a resource for potential biodiesel fuel users, government, and the public. In its Statement, the Association concludes:

1. All biodiesel fuel should meet specifications approved by ASTM International or comparable European standards-setting organizations.
2. Fuels blending up to 5% biodiesel with petroleum-based diesel fuel should not cause engine or fuel system problems.
3. Biodiesel blends may require additives to improve storage stability and allow use in a wide range of temperatures. In addition, the conditions of seals, hoses, gaskets, and wire coatings should be monitored regularly when biodiesel fuels are used.
4. There is limited information on the effect of pure biodiesel and biodiesel blends on engine durability during various environmental conditions.
5. Biodiesel fuels reduce emissions of hydrocarbons and carbon monoxide but increase nitrogen oxide emissions when compared to petroleum-based diesel fuel. Neither pure biodiesel fuel nor biodiesel blends should be used as a means to improve air quality in ozone non-attainment areas.
6. Individual engine manufacturers will determine the implications, if any, of the use of biodiesel fuels on their commercial engine warranties.

Copies of the complete statement are available on the EMA Web site at: www.enginemanufacturers.org. The Engine Manufacturers Association is a trade association representing worldwide manufacturers of internal combustion engines used in applications such as trucks and buses, farm and construction equipment, locomotives, marine vessels, and lawn, garden and utility equipment. EMA works with government and industry stakeholders to help the nation achieve its goals of cleaner fuels, more efficient engines and cleaner air." [39]

It is interesting to note that the Army has approved for a period of one year the use of Biodiesel B20 fuel in the non-deployable tactical schoolhouse vehicles at Fort Leonard Wood, Missouri. No emergency vehicles will be allowed to participate until a final report determines the impact of its use. [40]

SUMMARY AND RECOMMENDATIONS

A commercial item description (CID) for B20 biodiesel fuel blend has been issued that relies on blending materials meeting existing specifications for biodiesel and petroleum diesel, but also places requirements on the B20 biodiesel fuel blend.

The B20 biodiesel fuel blend is intended for use in all non-tactical diesel fuel consuming systems. There is ongoing research to determine more accurately the storage life of B20 biodiesel fuel blends. Until further data is available, it is recommended that the B20 biodiesel fuel blends in vehicle or storage tanks be used within six months of delivery, or sooner if possible. Fuels that have a TAN number greater than 0.25 mg KOH/g are not recommended for use. Other considerations for use of B20 biodiesel fuel are provided as notes for the user's consideration.

Various fleet tests using B20 biodiesel fuel blends have been completed or are in progress. The most commonly used biodiesel B100 in the U.S. has been methyl esters of soybean oil. This biodiesel tends to have the highest amount of unsaturation (double bonds), which can autoxidize to form acids, microparticulates and polymers. Polymers raise the viscosity of the biodiesel or biodiesel fuel blends. Other than minor repairable fuel system leaks and fuel filter plugging attributed to cleanup of dirty fuel systems, these field tests have been mostly uneventful. The NBB has estimated that over 40 fleets are operating on B20 biodiesel fuel blends in the U.S.

Previous evaluations of biodiesel and biodiesel blends have identified potential problem areas:

- Low temperature properties

- Storage stability
- Low compatibility with copper
- Incompatibility with nitrile rubber
- Degrade some filter media resulting in media migration
- Alter coalescing process for free water in water coalescer/separators

The adverse effect of biodiesel in B20 fuel blends on some fuel filters may be related to its solvency property, which could potentially dissolve filter binders, resins, and cause swelling of cellulosic fibers. Definitive laboratory evaluations of material compatibility of selected vehicle fuel system elastomers and fuel filters should be considered. Qualification procedures for approving fuel filters for use with biodiesel and biodiesel fuel blends should be considered.

Characterization data for biodiesel blends of biodiesel B100 samples in commercial diesel fuel obtained in Warren, MI, led to the following observations.

1. A number of companies in the U.S. can manufacture biodiesel (B100) from a variety of sources. Samples analyzed reflected at least six (6) different feedstocks; soybean, canola, cottonseed, used soybean, used cooking oil, and yellow grease.
2. Biodiesels can have properties that exceed the limits imposed on diesel fuel according to ASTM D 975. The concentration of biodiesel used in a final diesel-biodiesel blend will ultimately determine the level of impact to the diesel fuel blend.
3. The property that will be affected the most, and of greatest concern, is the cloud point. Biodiesels have a wide range of cloud points and their impact on the final blend must be carefully assessed. The results showed that for LS 2-D fuel blends, the cloud point increased around 2°C, however, for LS 1-D blends, the cloud point increased up to 20°C. This is a significant change that needs to be carefully controlled during winter operations. Investigation of other cold flow properties was outside the scope of this effort.
4. The PDSC (Figure 6) on B100 (used to assess oxidation stability) showed induction periods as low as 3 minutes, indicating some oxidation prone biodiesels. This low induction time of some biodiesels raises concern on the long term storage stability of the blended fuel.
5. No specific differences could be determined between the unused feedstock versus the used-feedstocks used in the manufacture of the biodiesel from the samples analyzed in this report. It appears that used feedstocks are more likely to result in biodiesels with higher cloud points than unused feedstocks.
6. The B20 blend is envisioned to be the highest concentration of biodiesel to be more commercially available in the USA. The B20 samples tested in this report showed that biodiesel blends, even at this high concentration of biodiesel, can meet a number of diesel fuel properties as defined by ASTM D975 but not the distillation 90% off temperature limit of 288°C for LS 1-D. The CID AA-59693 for B20 covers only LS 2-D B20 grade fuel, recognizing that a blend of LS 1-D and B20 will meet LS 2-D (but not LS 1-D) specification limits. Therefore, it is likely that lower biodiesel concentrations can meet the ASTM D 975 without any waivers or deviations. For the B100 biodiesel samples in this report, the concentration that can meet ASTM D 975 was between 5 and 10 percent. A project to develop data to characterize selected biodiesel samples and biodiesel (B2-B10) blends made with diesel fuels was initiated to support recommendations for inclusion of biodiesel in the ASTM D 975 specification for diesel fuel. Based on this data, it was apparent that inclusion of B5 biodiesel blend using both petroleum low sulfur diesel grades 1-D and 2-D could be transparent to the user if the biodiesel did not exceed 5 percent. For inclusion of B6-B20 biodiesel blends in ASTM D 975, it was recommended they be covered in a new annex to ASTM D 975.
7. Since B20 is the highest biodiesel blend concentration expected in the U.S. and it is the blend that can affect the final fuel properties, it is recommended that a B20 blend specification is needed in ASTM D975 or similar specification to ensure that the end user can assess and determine that the delivered product meets the needs of the equipment, including seasonal cold-weather changes.

Technical support should be provided to the Army one year use of Biodiesel B20 fuel in the non-deployable tactical schoolhouse vehicles at Fort Leonard Wood, Missouri. No emergency vehicles will be allowed to participate until a final report determines the impact of its use.

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APPENDIX A
Property Data for Biodiesel and Biodiesel Blends

Table A-1. B100 Biodiesel characterization summary

Feedstock	virgin soybean	virgin soybean	virgin soybean	virgin soybean	virgin soybean	recycled cooking oil	recycled cooking oil	recycled soybean cooking oil	virgin soybean	virgin vegetable oil blend	recycled vegetable oil blend	virgin canola	virgin cottonseed	yellow grease	virgin soybean
Sample #	FL-11235-00	FL-11237-00	FL-11250-00	FL-11257-00	FL-11262-00	FL-11271-00	FL-11272-00	FL-11273-00	FL-11274-00	FL-11291-00	FL-11292-00	FL-11316-01	FL-11317-01	FL-11318-01	FL-11341-01
KV @ 40°C, cSt (ASTM D 445)	4.092	2.883	4.13	4.085	5.851	4.657	4.96	5.023	3.991	4.0309	4.728	6.141	4.893	6.323	5.762
Total Water Content, ppm (ASTM D 4928)	316	1274	489	432	837	412	1216	1227	251	801	880	1443	1545	1757	1130
Cloud Point, °C (ASTM D 5773)	-1.6	3.1	-0.7	0.2	2.6	0.9	3.3	3.2	-2.4	0.0	4.8	-3.3	2.3	6.5	-1.4
Pour Point, °C (ASTM D 5949)	-3.0	0.0	0.0	0.0	-18.0	0.0	3.0	3.3	-3.0	-2.0	2	-6.0	0.0	2.0	-3
Freezing Point, °C (ASTM D 5972)	5.2	6.0	1.8	1.0	5.6	2.2	6.0	5.8	5.1	1.7	7.6	-0.3	6.4	9.1	0.8
Visual appearance (ASTM D 4176)	Pass (clear & bright)	Fail (sample is cloudy)	Pass (clear & bright)	Pass (clear & bright)	Fail (sample has particulate)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Fail (sample is cloudy)	Pass (clear & bright)
TAN, mg KOH/g (ASTM D 664)	0.1430	0.1584	0.1822	0.0701	0.1654	0.1820	0.3450	0.3867	0.1205	0.4093	0.3098	0.7612	0.3098	1.4888	0.6731
MCRT, mass % (ASTM D 4530)	0.0310	0.0502	0.0328	0.0438	0.0454	0.0484	0.0462	0.0151	0.0160	0.0319	0.0375	0.0795	0.0569	0.0829	0.1017
Calc. Iodine Value (AOCS 1c-85)	133.046	134.218	137.226	135.873	95.109	95.329	85.168	83.360	132.899	129.055	94.514	119.427	114.554	82.556	133.767
ASTM D 6186 modified (125°C), minutes	5.02	6.41	6.35	26.21	>65	>65	>65	>65	4.37	> 60	> 60	> 60	>83	> 60	5.20
Density, kg/L (ASTM D 1298)	0.8856	0.8872	0.8856	0.8864	0.8804	0.8817	0.8820	0.8828	0.8856	0.8856	0.8822	0.8906	0.8878	0.8861	0.8940

Table A-2. B20 LS 1-D Biodiesel Characterization Summary

Sample #	D1-11235.20	D1-11237.20	D1-11250.20	D1-11257.20	D1-11262.20	D1-11271.20	D1-11272.20	D1-11273.20	D1-11274.20	D1-11316.20	D1-11317.20	D1-11318.20	D1-11341.20	Diesel 1-D
KV @ 40°C, cSt (ASTM D 445)	1.949	1.958	1.947	1.951	2.100	1.999	2.017	2.028	1.942	2.100	2.015	2.115	2.085	1.639
Total Water Content, ppm (ASTM D 4928)	89.5	158.4	80.6	69.8	80.7	73.0	113.6	105.8	70.9	187.4	183.9	265.4	172.7	35.7
Cloud Point, °C (ASTM D 5773)	-24.7	0.5	20.4	-11	-19.5	21	-14.8	18.3	-25.1	-29.3	-1.9	-4.1	-22.9	-47.6
Pour Point, °C (ASTM D 5949)	-40.0	-30.0	-32.0	-32.0	-44.0	-32.0	-24.0	-32.0	-38.0	-39.8	-33.0	-27.0	-30.0	-62
Freezing Point, °C (ASTM D 5972)	-24.1	-1.5	-17.2	0.2	-15.0	-17.2	-8.0	-12.3	-22.7	-19.6	-4.3	3.2	-23.9	-46.3
Flash Point, °C	65	60	64	60	65	64	64	61	63	61	61	61	61	61
Distillation, °C (ASTM D 86)														
IBP	187.9	188.0	187.1	188.2	187.5	187.4	187.6	187.7	187.0	189.8	190.6	188.3	189.9	184.0
5%	199.7	200.5	199.4	200.1	199.3	200.0	200.0	199.5	199.8	200.0	199.8	201.0	200.0	198.8
10%	204.3	204.6	204.0	204.5	203.6	204.0	204.3	204.3	204.0	206.9	206.8	205.5	205.6	200.1
15%	208.1	208.1	207.7	208.2	207.4	207.9	207.9	208.0	208.1	210.6	210.6	209.3	209.4	205.3
20%	211.9	211.9	211.5	211.9	211.1	211.6	211.5	211.8	211.6	214.3	214.7	213.6	213.0	207.6
30%	219.0	218.8	218.6	218.4	217.9	218.7	219.0	218.8	218.6	220.5	220.9	220.4	219.8	213.1
40%	226.3	226.1	225.9	225.7	224.7	225.8	225.9	225.8	225.7	228.0	228.2	227.4	226.8	218.0
50%	234.4	234.4	234.1	233.7	232.5	233.8	233.6	233.7	233.8	236.5	236.3	235.4	235.0	223.1
60%	245.7	244.4	244.2	244.3	242.1	244.2	244.2	244.3	244.5	246.3	247.0	244.8	245.3	228.5
70%	260.1	259.8	259.7	259.2	258.2	260.4	260.0	259.0	259.9	262.1	263.0	260.2	262.0	234.4
80%	289.5	289.9	289.5	290.1	297.0	291.3	290.8	289.9	290.9	300.4	296.2	293.4	297.7	241.7
85%	313.3	313.6	313.4	313.3	337.9	315.5	314.0	313.8	314.2	324.3	318.4	318.6	322.4	245.9
90%	329.3	329.8	328.9	329.1	358.3	330.3	329.8	329.5	329.3	338.4	331.5	334.4	336.5	251.3
95%	336.2	336.2	335.7	335.7	368.9	336.9	336.8	336.9	335.4	348.3	342.3	346.3	347.3	259.2
End Point	341.4	342.4	341.8	342.0	372.7	342.9	343.5	345.2	340.3	359.6	350.6	354.0	359.4	276.4
Visual appearance (ASTM D 4176)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)			Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)
TAN, mg KOH/g (ASTM D 664)	0.0589	0.0497	0.0385	0.0175	0.0449	0.0870	0.0764	0.0764	0.0309	0.1395	0.0554	0.3056	0.1409	0.0014
MCRT, mass % (ASTM D 4530)	0.0000	0.0051	0.0061	0.0021	0.0075	0.0077	0.0083	0.0095	0.0024	0.0168	0.0135	0.0178	0.0239	0.0000
ASTM D 6217 (GF/F), mg/L	0.6	4.2	2.0	0.6	2.8	7.4	1.0	2.0	0.8	1.2	1.6	2.8	1.8	0.4
Density, kg/L (ASTM D 1298)	0.8334	0.8334	0.8329	0.8329	0.8324	0.8324	0.8322	0.8324	0.8324	0.8334	0.8337	0.8329	0.8344	0.8199

Table A-3. <u>B20 LS 2-D Biodiesel Characterization Summary</u>

Sample #	D2-11235.20	D2-11237.20	D2-11250.20	D2-11527.20	D2-11262.20	D2-11271.20	D2-11272.20	D2-11273.20	D2-11274.20	D2-11316.20	D2-11317.20	D2-11318.20	D2-11341.20	Diesel 2-D
KV @ 40°C, cSt (ASTM D 445)	2.882	2.899	2.887	2.896	3.137	2.977	2.991	3.015	2.878	3.142	2.988	3.153	2.913	2.886
Total Water Content, ppm (ASTM D 4928)	93.5	200.8	85.2	81.5	114.8	87.2	149.7	137.4	88.9	194.2	194.5	239.3	226.2	41.4
Cloud Point, °C (ASTM D 5773)	-13.7	-12.4	-12.5	-12.3	-11.9	-12.1	-11.5	-11.5	-13.6	-13.4	-9.1	-9.2	-12.8	-15.2
Pour Point, °C (ASTM D 5949)	-22.0	-24.0	-22.0	-20.0	-20.0	-20.0	-18.0	-16.0	-20.0	-18.0	-18.0	-21.8	-18.0	-28.0
Freezing Point, °C (ASTM D 5972)	-11.6	-10.2	-10.4	-10.2	-9.0	-11.0	-9.3	-9.4	-11.6	-9.9	-9.9	2.3	-9.7	-11.7
Flash Point, °C	63	55	62	58	60	60	60	57	61	65	65	61	64	56
Distillation, °C (ASTM D 86)														
IBP	190.7	190.0	189.8	188.6	188.6	187.9	188.7	187.8	189.2	190.8	189.8	189.3	188.5	183.7
5%	215.2	216.1	215.7	215.4	215.0	214.9	215.1	214.5	215.8	218.4	215.1	216.0	217.4	211.1
10%	226.4	227.5	227.3	227.3	226.8	226.8	226.5	226.8	227.5	229.9	229.4	230.2	229.8	220.2
15%	235.5	234.7	234.2	233.9	233.6	234.2	234.2	234.2	234.3	237.5	236.7	238.3	236.6	227.2
20%	241.9	242.2	241.9	241.7	241.4	242.0	241.9	241.8	241.4	245.1	244.3	245.3	244.7	232.6
30%	256.6	256.5	256.7	256.8	255.6	257.3	256.9	256.8	256.8	259.8	259.0	259.9	259.2	244.9
40%	270.3	270.5	270.8	270.6	269.5	271.0	270.7	270.6	270.6	273.5	273.1	273.5	273.1	255.6
50%	283.4	284.1	284.0	283.8	283.5	284.5	284.0	283.7	283.8	287.0	286.2	286.8	286.2	265.6
60%	297.0	297.7	297.6	267.6	299.4	298.0	297.5	297.3	297.6	300.9	299.5	300.4	3001.0	276.5
70%	310.5	311.0	311.2	310.9	316.8	311.6	311.0	310.7	311.2	314.8	312.6	314.7	313.6	287.8
80%	323.4	323.7	323.4	323.2	336.3	324.0	323.7	323.2	323.4	328.2	325.2	327.9	326.1	301.6
85%	329.1	329.2	328.8	328.8	346.4	329.6	329.4	329.0	328.8	334.8	331.2	334.6	332.9	309.9
90%	334.7	334.6	334.3	334.2	356.6	335.2	335.1	334.8	334.1	343.1	338.0	343.7	341.1	320.6
95%	343.6	342.3	341.7	341.3	368.4	342.8	343.9	344.0	341.1	355.1	354.2	354.5	349.5	338.0
End Point	347.1	353.3	351.8	353.8	375.4	352.6	353.2	354.1	351.4	364.0	359.0	362.9	360.6	357.4
Visual appearance (ASTM D 4176)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)	Pass (clear & bright)
TAN, mg KOH/g (ASTM D 664)	0.1108	0.0722	0.0442	0.0147	0.0533	0.0428	0.0785	0.0799	0.0358	0.1402	0.0533	0.2938	0.1332	0.0014
MCRT, mass % (ASTM D 4530)	0.0104	0.0122	0.0114	0.0057	0.0134	0.0135	0.0169	0.0177	0.0074	0.0186	0.0135	0.0227	0.0296	0.0000
ASTM D 6217 (GF/F), mg/L	0.8	14.8	2.2	0.8	3.0	7.4	1.2	2.2	1.0	2.0	2.8	8.6	4.0	0.6
Density, kg/L (ASTM D 1298)	0.8735	0.8687	0.8682	0.8682	0.8687	0.8676	0.8685	0.8679	0.8682	0.8692	0.8687	0.8687	0.8698	0.8639